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AN EXAMINATION OF RADIOACTIVITY IN THE CRUSHED ROCK FROM THE PM--ETC(U)

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AN EXAMINATION OF RADIOACTIVITY
IN THE CRUSHED ROCK FROM THE
PM-3A NUCLEAR REACTOR SITE,
McMURDO, ANTARCTICA

by

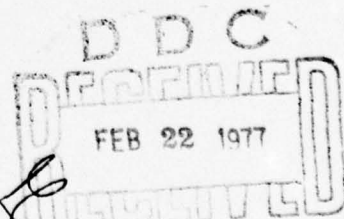
Herbert V. Weiss

Undersea Sciences Department

January 1977



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AN ACTIVITY OF THE NAVAL MATERIAL COMMAND

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This work was performed from October to December 1976 under Naval Nuclear Unit Project No 537 A-76-WR-T 0004 by H. V. Weiss of the Chemistry and Environmental Sciences Group.

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 14 NUC-TP-551	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER 9
4. TITLE (and Subtitle) 6 AN EXAMINATION OF RADIOACTIVITY IN THE CRUSHED ROCK FROM THE PM-3A NUCLEAR REACTOR SITE, McMurdo, ANTARCTICA.	5. FUNDING NUMBERS Research and Development October-December 1976 rept.	
7. AUTHOR(s) 10 Herbert V. Weiss	8. CONTRACT OR GRANT NUMBER(s)	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Undersea Center San Diego, California 92132	10. PROGRAM ELEMENT PROJECT, TASK AREA & WORK UNIT NUMBERS N0537A-76-WR-T0004	
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Nuclear Power Unit Port Hueneme, California	12. REPORT DATE 11 December 1976	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	13. NUMBER OF PAGES 13 12/36p	
	15. SECURITY CLASS. (of this report) Unclassified	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Radioactivity Nuclear reactor Strontium-90 Yttrium-90		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Cs 137 Sr 90 → Radioactivity in McMurdo rock consists primarily of ¹³⁷ Cs and ⁹⁰ Sr. The 6500 yd ³ of affected material is estimated to contain 2.7×10^{-1} and 3.1×10^{-2} Ci of these two ele- ments, respectively. Sr 90 0.27 0.031 Cs 137 → ¹³⁷ Cs, but not ⁹⁰ Sr, is tenaciously bound to the rock in seawater. Both are held firmly by the rock in distilled water, thus, release to the environment would be significantly less in terrestrial rather than marine disposal.		

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SUMMARY

Problem: In the operation of the PM-3A nuclear power reactor at McMurdo, Antarctica, a small amount of radioactivity was dispersed into the immediate environment. Such discharge is common to all nuclear power reactors and the content of the discharges was within the limits established by the Nuclear Regulatory Commission. The Navy plans to remove the affected substrate. The volume of substance involved is 6500 yd³, and the potential disposal sites under consideration are marine or terrestrial.

Accordingly, a study was undertaken to characterize the types and quantity of radioactivity and to develop a sense of the degree to which the radioactivities are mobilized by seawater and distilled water.

Results: Radioactivity in McMurdo rock consists primarily of ¹³⁷Cs and ⁹⁰Sr. Traces of ¹³⁴Cs and ⁶⁰Co are also detectable.

The 6500 yd³ of radioactive material is estimated to contain 2.7×10^{-1} Ci of ¹³⁷Cs and an upper limit of 3.1×10^{-2} Ci of ⁹⁰Sr.

The ¹³⁷Cs, but not ⁹⁰Sr, is tenaciously bound to the rock upon dynamic exposure to seawater. Both radionuclides are firmly held to the rock in the presence of distilled water. From the results of column experiments it is estimated that less than 1 percent of ¹³⁷Cs and only several percent of ⁹⁰Sr would be mobilized upon exposure to 100 inches of rainfall.

The greatest fraction of radioactivity is associated with smaller particles and these, in turn, represent a small fraction of the total mass. Further, radioactivity associated with the larger particles was substantially relieved by air blasting or flotation; both actions remove adhering fine particles. Perhaps the logistic problem and ultimate disposal can be minimized by some simple cleansing operation such that a large fraction of the material could be released to the general environment.

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INTRODUCTION

In the course of operating the PM-3A plant at McMurdo, Antarctica, a small amount of radioactivity was discharged to the environment (Ref. 1). Such discharge is common to all nuclear power reactors and the radioactive content of the discharges is within the limits established by the Nuclear Regulatory Commission. The Navy plans to remove the affected substrate.

The affected material consists of approximately 6500 yd³ of crushed rock. A representative sample was acquired on 8 October 1976. The sample was studied with a view toward characterizing its radionuclide composition, especially with regard to the longer lived fission products, ¹³⁷Cs and ⁹⁰Sr. Further it was submitted to various treatments for evaluation of the probable disposition of the radionuclides as a function of terrestrial or oceanic disposal, the alternatives under consideration. The treatments consisted of equilibrating the rock with either seawater or distilled water and measuring the distribution of radioactivity in the solid and liquid phases. Uptake of Cs and Sr radioelements from water by the rocks was also examined to gauge the relative affinity of the two phases for these elements. The more recent examinations emphasized the behavior of the radioactivities in a terrestrial environment. Since in such an environment the greatest mobilizing force is precipitation, the leachability of the radionuclides from these rocks by distilled water was studied.

This report describes the methodology and results of the examinations performed and provides a measure of the degree of translocation of ¹³⁷Cs and ⁹⁰Sr in a land disposal site.

The sample of crushed rock (3 kg) is characterized as a fine-grained vesicular olivine basalt (Ref. 2). The material ranges in size from several centimeters to very fine particulates. The density of the material is 2.5 g/cm³.

EXPERIMENTAL METHOD

INSTRUMENTATION

Gamma ray spectral analysis was performed with a sodium iodide flat- or well-type crystal detector coupled to a 400-channel pulse height analyzer (Technical Measurements Corporation). Gross gamma ray measurements of ⁸⁵Sr were made with a 3-inch well-type sodium iodide detector connected to a scaler. The beta rays of ⁹⁰Y were counted with an automatic Beckman Wide Beta instrument, whose background was about 1.4 counts per minute.

PARTICLE SIZE SEPARATION

In conformity with the NBS procedure (Ref. 3), the material studied in the first phase of this work was that which passed through a 20-mesh wire screen to deliver particles less than 0.840 mm. This fraction will be referred to as "20 mesh." The 20-mesh fraction was submitted to gamma-ray and ^{90}Sr analyses, a study of the distribution of radionuclides upon mixing with seawater or distilled water and of the uptake of ^{137}Cs and ^{85}Sr from these media.

Owing to the wide distribution of particle size it appeared worthwhile to examine the level of ^{137}Cs activity as a function of size. Accordingly the sample was fractionated into seven sizes by passage through screens of 4, 12, 16, 20, 60 and 250 mesh to give the following distribution: > 4.760 , $1.680-4.760$, $1.130-1.680$, $0.840-1.130$, $0.234-0.840$, $0.061-0.234$ and < 0.061 mm. For expedience these fractions are assigned numbers 1 to 7 in the order given. The fractions were analyzed for their ^{137}Cs content as well as the leachability of this nuclide and of ^{85}Sr from them.

RADIOMETRIC ANALYSES

^{137}Cs Measurement

^{137}Cs was determined instrumentally by pulse height analysis. Weighed samples (50 g) were deposited in 250-ml Erlenmeyer flasks and analyzed on the flat detector. Liquids, 25 ml in volume, were contained within 50-ml centrifuge tubes and measured with the well crystal.

By Covell's method of integration (Ref. 4), the photopeak response to a ^{137}Cs standard (Amersham-Searle) was determined under counting conditions for solid and liquid samples. The respective counting efficiencies were 5.16 and 8.46 percent.

The background in the photopeak area with the well crystal was 146 counts/100 min. The standard error computed (Ref. 6), for this count was ± 150 . Background correction on the solid crystal was insignificant compared with the solid sample counting rates and none was made.

^{90}Sr - ^{90}Y Analysis

Crushed Rock. In duplicate analyses, 10 g of 20-mesh crushed rock were leached with 50 ml hot nitric acid under reflux for 1 hour. The liquid was collected by centrifugation. The sediment was washed with water and the water washings were combined with the acid leachate. Forty mg of Sr carrier and a known quantity of ^{85}Sr were added to the solution. Sr was separated from Ca by the Willard and Goodspeed method.

Water. To either seawater or distilled water which had been exposed to and subsequently separated from the rock sample were added Sr carrier and ^{85}Sr tracer as above. Sufficient sodium carbonate was added to fully precipitate Ca and Sr from solution. After separation

of the carbonates by centrifugation the solid was dissolved with several ml of 2 N HNO_3 . The Ca-Sr separation was effected as above.

At this point the samples, whether derived from soil or water, were susceptible to the same radiochemical purification steps. The procedure described by Shipman (Ref. 5) was followed. The purification consisted of iron scavenging, barium chromate precipitation, and strontium carbonate precipitation. After dissolution of the carbonate, yttrium was milked from solution by hydroxide precipitation. Upon separation of yttrium, the solution was acidified and stored for 3 weeks. Yttrium was again milked as the hydroxide, converted to the oxalate, dried, weighed, and mounted on brass. The ^{90}Y was counted at least twice daily for 9 days. The ^{85}Sr content of the solution subjected to the second milking was measured.

The ^{90}Y counting data were corrected for a long-lived tail, decay, ^{85}Sr yield, yttrium carrier yield and counting efficiency.

A counting efficiency of 52 percent was determined for a ^{90}Sr - ^{90}Y standard (Amersham-Searle) after milking the ^{90}Y in the manner described for samples and comparing its counting rate with the known disintegration rate.

Procedures

Equilibration Study. The 20-mesh sample was separated into 50-g aliquots and the gamma ray spectrum of each was acquired.

One hundred ml of freshly filtered seawater were added to each of two aliquots. Two additional aliquots received this volume of distilled water. The mixtures were shaken continuously for 24 hours. The substrate was separated from solution by centrifugation. The supernatant was filtered through 0.45- μ membrane filters to remove fine particulates. To the clear filtrate were added 10-20 mg CsCl to avoid surface adsorption. The solution was reduced to a small volume, transferred to a tube, and then diluted to the standard counting volume.

Substrates were returned to their original containers, re-equilibrated with seawater or distilled water, and treated as above. Rock samples that received seawater were processed through four complete cycles and those equilibrated with distilled water through either two or three cycles. (In extractions with distilled water, separation of the fine particles becomes increasingly more difficult with successive cycles, to the point of impracticality.)

The aqueous fractions were analyzed for their ^{137}Cs content as described.

To study the equilibration of ^{90}Sr , 10 g of 20-mesh sample were used. Each of three samples was equilibrated for a single 24-hour cycle with either seawater or distilled water. After separation of phases, the aqueous fraction was analyzed for ^{90}Sr .

Uptake Study. To gain understanding of the affinity of the rock sample for the Cs and Sr radionuclides, an uptake experiment was performed. In each of four flasks, 10 g of 20-mesh rock were placed. Each received 75 ml of seawater or distilled water and ^{137}Cs or ^{85}Sr , and none of the combinations was duplicated. The mixtures were shaken for 24 hours, separated by centrifugation, and the solid was gross gamma counted. Solids were subsequently spiked with known quantities of the respective radioactivities and re-counted. From these data the fraction of activity removed from solution by the rock sample was computed.

^{137}Cs and Particle Size. Each of the size-fractionated samples was submitted to ^{137}Cs analysis to determine the relationship between ^{137}Cs activity and particle size.

Compressed Air and Flotation Treatment. To evaluate whether larger particles could be relieved of some of their radioactivity burden, two treatments were applied. A portion of Fraction 1, while supported on wire screen, was freed of small particulates adhering to surfaces with blasts of compressed air. Particles from Fraction 2 were agitated for several seconds in a column of distilled water. Several seconds later, after the larger particles had settled, the aqueous phase containing the unsettled fine particles was decanted. This flotation process was repeated several times. The efficiency of these treatments in diminishing the radioactive content was determined by ^{137}Cs gamma spectral analysis.

Column Experiment. Each of a set of 12-mm-I.D. glass columns was filled with 20-50 g of crushed rock of one of the seven particle size fractions. A volume of distilled water (250 ml) was passed through the column at a flow rate not in excess of 2 ml/min. Owing to time constraints, only 50 ml was passed through the column that contained particles from Fraction 7. The flow rate for this column was only 0.035 ml/min. The effluent was filtered through a membrane filter, evaporated to 25 ml in the presence of Cs carrier and pulse height analyzed for ^{137}Cs .

Each column, except for that containing Fraction 5, was then treated with about 15 ml of a slightly ammoniacal solution containing a known quantity of ^{85}Sr . The column with Fraction 5 was spiked with acidic ^{85}Sr solution that had not been alkalinized. Within several minutes elution of the columns with 250 ml distilled water was begun. Each 25-ml aliquot was collected separately and analyzed for ^{85}Sr . The columns containing Fractions 1 and 2 were eluted with an additional 250 ml of distilled water 4 days after the initial elution. Each eluate was collected as a composite, and a 25-ml aliquot of each was again analyzed for ^{85}Sr tracer.

RESULTS

In the analysis of gamma-ray spectra of six 50-g aliquots of the 20-mesh sample a value of 110.0 ± 6.9 d/m ^{137}Cs per gram was determined. These spectra also revealed the presence of ^{134}Cs and ^{60}Co , although in considerably smaller quantity.

In equilibration experiments (Table 1) with seawater, 2.58 ± 0.18 , 0.85 ± 0.23 , 1.08 ± 0.02 , and 1.00 ± 0.45 percent of ^{137}Cs were removed from the rock in four successive extractions. In distilled water 0.62 ± 0.02 percent of this radionuclide was displaced from the substrate in the first cycle. In subsequent cycles the count rate was below the level of statistical significance (Table 1).

The fraction of crushed rock assayed in duplicate for ^{90}Sr gave an average disintegration rate of 5.4 ± 0.1 d/m/g. In the equilibration study 62.8 ± 1.4 percent was displaced from the rock to the seawater matrix. In the distilled water extracts this nuclide was detected to the extent of 1.1 percent.

The uptake of ^{137}Cs and ^{85}Sr by rock from seawater and distilled water appears in Table 2. The extent of incorporation with the solid substrate was 96.5 and 88.3, respectively, for the two sources in distilled water. The redistribution of ^{85}Sr is not so pronounced in seawater; the corresponding values are 85.3 and 35.5 percent.

^{137}Cs associated with size-fractionated rock appears in Table 3. The activity level is inversely related to particle size. Thus only 17.3 percent of the activity resides with the two largest fractions, whose mass accounts for 55.1 percent of the total sample weight. The corresponding values for the three fractions of smallest particle size are 65.1 and 30.0 percent.

In the column experiment, ^{137}Cs was undetectable in the effluents derived from each of the columns. The weight of the size-fractionated rock packed in each column and the detection limit, which is defined as a value two times greater than the photopeak background (Ref. 6), (146 counts per 100 min), appear in Table 4-A.

The elution characteristics of ^{85}Sr from the same columns are shown in Table 4-B. For columns that contained Fractions 1, 2 and 5, the values for ^{85}Sr are relatively high in the first fractions of eluate; with successive fractions a relatively constant value is attained, and the values are always a fraction of a percent. The average quantity of ^{85}Sr in the second elution, which occurred 4 days later with columns of Fraction 1 and 2, was not markedly different from the last fractions of the preceding elution.

When the surfaces of the rock of largest size were freed of fine particulates by a jet of air, the ^{137}Cs level dropped by 57 percent. Separation of the fine particles in Fraction 2 from the bulk by flotation diminished the gamma ray activity by 62 percent.

DISCUSSION

From the analysis of the various particle sizes for ^{137}Cs content, the amount of this radioactivity associated with the 6500 yd^3 of affected crushed rock can be estimated. This quantity is 2.7×10^{-1} Ci.

A comparable calculation is not possible for ^{90}Sr since only the 20-mesh sample was analyzed. However, this fraction, which represents 30 percent of the sample weight, accounts for the bulk of gamma ray activity. This relationship probably also holds for ^{90}Sr . Thus scaling up the ^{90}Sr assay to the total mass of rock provides an upper limit of concentration. This value is 3.1×10^{-2} Ci.

The equilibration studies point to the tenacity with which ^{137}Cs is retained by the rock even in seawater. Only small fractions of the activity are extracted into the aqueous phase under dynamic experimental conditions. ^{90}Sr , on the other hand, is released at a considerably more rapid rate in marine waters. These studies suggest that exposing the rocks to rainwater would not produce rapid release of either activity. The results of the uptake studies further support these conclusions since the equilibrium is in favor of interaction and association with the rock, especially in the distilled water medium.

The column experiments were performed to estimate the effect of leaching by rainwater in case the rock were to be deposited terrestrially. The column dimension was such that percolation of 250 ml of solution through it represented leaching by 100 inches of water. Were the disposal to occur in an environment such as Port Hueneme, where the annual rainfall is about 10 inches, the laboratory conditions simulate a 10-year exposure. With respect to ^{137}Cs the results indicate that less than 1 percent would be mobilized in this period of time.

In the ^{85}Sr tracer column experiment, the relatively high activity levels in the earliest fractions of some of the columns represent tracer that had not undergone adsorption upon its placement on the column.

Fractions 1 and 2 were of the largest particle sizes and therefore possessed the smallest surface area for interaction with ^{85}Sr . In addition the particles were of such dimension and irregularity that considerable dead space existed within each of these columns, and this effect was more pronounced with Fraction 1. A comparison of the ^{85}Sr activity in the first fractions of eluate for these two columns clearly shows the influence of column packing with the procedure that was followed.

The relatively greater activity level of the earliest fractions of eluate from the column with Fraction 5 as compared with its neighbors points to the effect of placing a slightly acid tracer solution on the column. It is well recognized that the adsorption efficiency of elements diminishes as the hydrogen-ion concentration increases.

In all cases by the time the fifth fraction of the elution volume passed through, a steady rate of tracer discharge was evident. The activity level in the second 250-ml elution of columns containing particles of the two largest sizes is consistent with the latter fractions of the preceding elution. This observation, coupled with the fact that the elutions occurred 4 days apart, supports the notion that an equilibrium condition was attained under the experimental conditions devised. Thus, the behavior of ^{85}Sr at the relatively constant rate of discharge probably parallels that of ^{90}Sr . Accordingly, the leaching of perhaps several percent of ^{90}Sr may be expected should the affected McMurdo rock be exposed to 100 inches of rainfall.

The total absence of measurable amounts of ^{85}Sr in the eluate of Fraction 7 may be related to extremely slow elution rate (0.035 ml/min) or perhaps to a greater exchange capacity of this fraction compared with larger particles.

The activity level of the Fractions 1 and 2 was diminished substantially by fine particle removal either through the action of an air jet or by flotation. Perhaps some such expedient could be used to reduce the level of radioactivity to an extent that retention of a sizable fraction of rock at McMurdo would be acceptable.

SUMMARY

Radioactivity in McMurdo rock consists primarily of ^{137}Cs and ^{90}Sr . Traces of ^{134}Cs and ^{60}Co are also detectable.

The 6500 yd^3 of radioactive material is estimated to contain 2.7×10^{-1} Ci of ^{137}Cs and an upper limit of 3.1×10^{-2} Ci of ^{90}Sr .

The ^{137}Cs , but not ^{90}Sr , is tenaciously bound to the rock even upon dynamic exposure to seawater. Both radionuclides are firmly held to the rock in the presence of distilled water. From the results of column experiments it is estimated that less than 1 percent of ^{137}Cs and only several percent of ^{90}Sr would be mobilized upon exposure to 100 inches of rainfall.

The greatest fraction of radioactivity is associated with smaller particles and these, in turn, represent a small fraction of the total mass. Further, radioactivity associated with the larger particles was substantially relieved by air blasting or flotation; both actions remove adhering fine particles. Perhaps the logistic problem and ultimate disposal can be minimized by some simple cleansing operation such that a large fraction of the material could be released to the general environment.

Table 1.

^{137}Cs AND ^{90}Sr EXTRACTED INTO SEAWATER OR DISTILLED WATER
AFTER 24-HOUR EQUILIBRATIONS WITH 20-MESH ROCK

		Extracted into Aqueous Phase (%)			
		Cycle			
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>Seawater</u>	^{137}Cs	$2.58 \pm 0.18^*$	0.85 ± 0.23	1.08 ± 0.02	1.00 ± 0.45
	^{90}Sr	62.8 ± 1.4			
<u>Distilled Water</u>	^{137}Cs	0.62 ± 0.02	$(0.09 \pm 0.04)^{**}$	(0.01)	
	^{90}Sr	1.1 ± 0.0			

*The values presented are averages of at least two determinations; the attendant error is the average deviation from the mean.

**Bracketed values are not statistically significant. The limit of detection is defined as a value equivalent to twice background and in this case the limit is 0.4%.

Table 2.

UPTAKE OF ^{137}Cs AND ^{85}Sr FROM SEAWATER
AND DISTILLED WATER BY 20-MESH ROCK SAMPLE

Radio Element	Activity Associated with Rock (%)	
	Seawater	Distilled Water
^{85}Sr	35.5	88.3
^{137}Cs	85.3	96.5

Table 3.

ROCK SIZE DISTRIBUTION AND
 ^{137}Cs CONTENT IN THE VARIOUS FRACTIONS

Fraction	Particle Size (mm)	Weight (g)	^{137}Cs		Fraction of Total (%)	
			d/min/g	Ci/g 10^{-12}	Weight	^{137}Cs
1	> 4.760	993	9.6	4.4	33.9	6.8
2	1.680-4.760	623	23.7	10.8	21.2	10.5
3	1.130-1.680	237	41.7	19.0	8.1	7.0
4	0.840-1.130	200	74.9	34.0	6.8	10.7
5	0.234-0.840	394	69.1	31.4	13.4	19.4
6	0.061-0.234	357	111.0	50.4	12.2	28.2
7	< 0.061	129	190.2	86.4	4.4	17.5
Weighted Mean			47.9	21.7		

Table 4.

ELUTION OF ^{137}Cs AND ^{85}Sr FROM COLUMNS (12 mm I.D.)
PACKED WITH VARIOUS PARTICLE SIZE FRACTIONS

A. ^{137}Cs

<u>Fraction</u>	<u>Packing Weight (g)</u>	<u>^{137}Cs Eluted</u>	<u>Detection Limit**(%)</u>
1	50	N.D. *	3.6
2	50	N.D. *	1.5
3	50	N.D. *	0.8
4	50	N.D. *	0.5
5	50	N.D. *	0.5
6	40	N.D. *	0.4
7	20	N.D. *	0.5

* N.D. — None detected

** Detection Limit (%) is the count rate equal to twice the photopeak background count (146 counts/100 min) divided by the number of ^{137}Cs counts in the column times 100.

B. ^{85}Sr ^{85}Sr IN EACH 25 ML FRACTION ELUATE (%)

<u>Fraction</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>Composite 11-20</u>
1	31.2	5.9	2.2	1.0	0.50	0.48	0.30	0.50	0.50	0.10	0.20
2	5.8	0.36	0.36	—	0.50	0.12	0.25	0.20	0.16	0.08	0.05
3	0.11	0.26	0.09	0.12	0.10✓	0.10✓	0.10✓	0.10✓	0.10✓	0.10✓	
4	0.44	0.23	0.07	0.04	0.03	0.02	0.06	0.06	0.03	0.11	
5	3.3	0.40	0.50	0.33	0.01	0.03	0.03	0.02	N.D.	0.03	
6	0.15	0.06	0.05	0.08	0.09	0.24	0.12	0.13	0.13	0.08	
7	N.D.	N.D.	N.D.								

✓ Average of six 25-ml-fraction composite

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